

FABRIC SOFTENER COMPOSITIONS

FIELD OF THE INVENTION

The present invention relates to the use of fabric softener compositions comprising selected polyorganosiloxanes, or mixtures thereof, together with selected additives for the wrinkle recovery or reduction of wet soiling in domestic applications. In particular it relates to textile softening compositions for use in a textile laundering operation to impart excellent wrinkle recovery and wet soiling benefits on the textile.

BACKGROUND OF THE INVENTION

Wrinkles in fabrics are caused by the bending and creasing of the textile material which places an external portion of a filament in a yarn under tension while the internal portion of that filament in the yarn is placed under compression. Particularly with cotton fabrics, the hydrogen bonding that occurs between the cellulose molecules contributes to keeping wrinkles in place. The wrinkling of fabric, in particular clothing, is therefore subject to the inherent tensional elastic deformation and recovery properties of the fibres which constitute the yarn and fabrics.

There is a demand for a quick fix which will help to diminish the labour involved in home laundering and/or the cost and time involved in dry cleaning or commercial laundering. This has brought additional pressure to bear on textile technologists to produce a product that will sufficiently reduce wrinkles in fabrics, especially clothing, and to produce a good appearance through a simple, convenient application of a product.

The present invention helps remove wrinkles from fabrics, including clothing, dry cleanable fabrics and draperies, without the need for ironing. The present invention can be used on washed clothing, which is damp or dry, to relax wrinkles and give clothes a ready to wear look that is demanded by today's consumer. The present invention also essentially eliminates the need for touch up ironing usually associated with closet, drawer, and suitcase storage of garments.

- 2 -

When ironing is desired however, the present invention can also act as an excellent ironing aid. The present invention makes the task of ironing easier and faster by creating less iron drag. When used as an ironing aid, the composition of the present invention produces a crisp, smooth appearance similar to that of spray starch ironing aids without the dry residue or flaking that occurs with typical spray starch ironing aids. It appears that recognition of improved "ease of ironing" can arise from a combination of at least three factors, namely fewer wrinkles to be removed, wrinkles more easily removed (e.g. with less weight upon the iron), or more completely removed, and less effort required to slide the iron along the fabric.

An additional benefit of the composition of the present invention is an in-wear wrinkle control benefit. The composition of the present invention can help to prevent future wrinkles from forming in the fabric even after the fabric has been through a wash cycle, or a tumble drying process.

Another property which it would be desirable to impart is the reduction of wet soiling. Some wrinkle recovery treatments have the disadvantage that wet soiling is adversely effected. Hence, it is often also necessary to treat the textile material further to improve its wet soiling characteristics. It would therefore be desirable to find a process according to which wrinkle recovery as well as wet soiling is improved.

Surprisingly, it has been found that the use of selected polyorganosiloxanes, or mixtures thereof, together with selected additives in fabric softener compositions provide excellent wrinkle recovery and ironing improvement effects as well as a reduction of wet soiling when applied to fabrics during a textile laundry operation.

As given above one component of the compositions of the present invention are polyorganosiloxanes. Such compounds are known to be used on an industrial scale to finish fabrics by providing them with a permanent or semi-permanent finish aimed at improving their general appearance. Significant for these industrial fabric finishing processes is a co-called curing step generally involving temperatures in excess of 150°C often for periods of one hour or more. The object here is to form a chemical finish which resists destruction during subsequent cleaning/laundrying of fabrics. This process of finishing is not carried out in domestic applications and accordingly one would not expect benefits of a comparable nature or magnitude from polyorganosiloxanes included as adjuncts in domestic softeners.

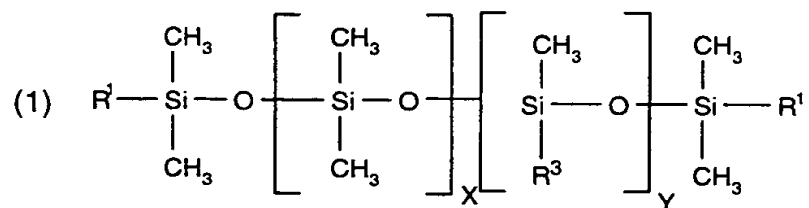
- 3 -

Indeed, it is noteworthy that if the compounds of the current invention achieved a permanence associated with industrial textile finishing, problems associated with a cumulative build through the wash cycles could occur such as fabric discoloration and even in extremes an unpleasant feel to the wearer.

SUMMARY OF THE INVENTION

This invention relates to a method of use of a fabric softener composition for the wrinkle recovery treatment or the reduction of wet soiling of textile fibre materials in domestic applications, which softener composition comprises:

- A) a fabric softener;
- B) at least one additive selected from the group consisting of
 - a) a polyethylene, or a mixture thereof,
 - b) a fatty acid alkanolamide, or a mixture thereof,
 - c) a polysilicic acid, or a mixture thereof, and
 - d) a polyurethane, or a mixture thereof; and
- C) a dispersed polyorganosiloxane of formula (1)



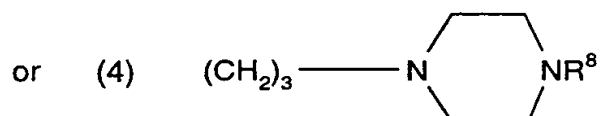
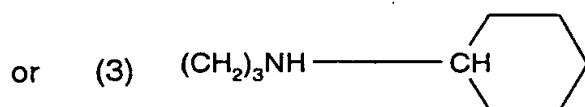
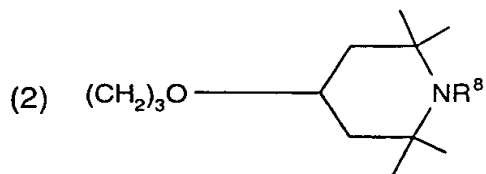
wherein

R¹ is OH, OR² or CH₃

R² is CH₃ or CH₂CH₃

R³ is C₁-C₂₀alkoxy, CH₃, CH₂CHR⁴CH₂NHR⁵, or CH₂CHR⁴CH₂N(COCH₃)R⁵

- 4 -



R^4 is H or CH_3

R^5 is H, $\text{CH}_2\text{CH}_2\text{NHR}^6$, $\text{C}(=\text{O})-\text{R}^7$ or $(\text{CH}_2)_z-\text{CH}_3$

z is 0 to 7

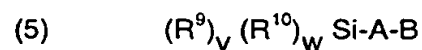
R^6 is H or $\text{C}(=\text{O})-\text{R}^7$

R^7 is CH_3 , CH_2CH_3 or $\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$

R^8 is H or CH_3

the sum of X and Y is 40 to 4000;

or a dispersed polyorganosiloxane which comprises at least one unit of the formula (5)



wherein

R^9 is CH_3 , CH_3CH_2 or Phenyl

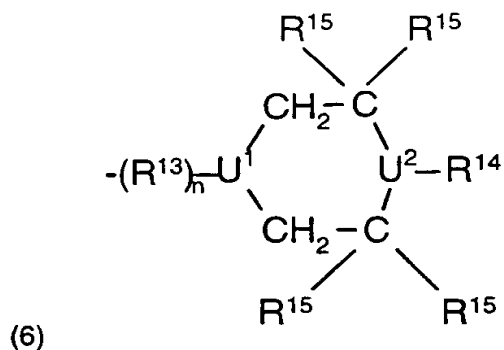
R^{10} is $-\text{O}-\text{Si}$ or $-\text{O}-\text{R}^9$

the sum of v and w equals 3, and v does not equal 3

$\text{A} = -\text{CH}_2\text{CH}(\text{R}^{11})(\text{CH}_2)_k$

$\text{B} = -\text{NR}^{12}((\text{CH}_2)_l-\text{NH})_m\text{R}^{12}$, or

- 5 -



n is 0 or 1

when n is 0, U¹ is N, when n is 1, U¹ is CH

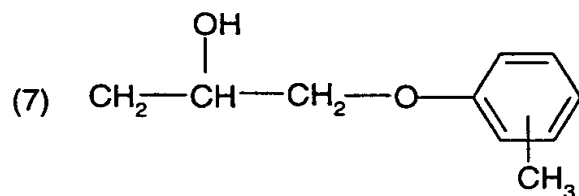
l is 2 to 8

k is 0 to 6

m is 0 to 3

R¹¹ is H or CH₃

R¹² is H, C(=O)-R¹⁶, CH₂(CH₂)_pCH₃ or



p is 0 to 6

R¹³ is NH, O, OCH₂CH(OH)CH₂N(Butyl), OOCN(Butyl)

R¹⁴ is H, linear or branched C₁-C₄ alkyl, Phenyl or CH₂CH(OH)CH₃

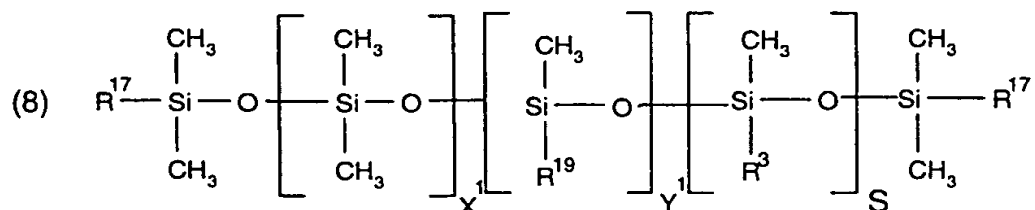
R¹⁵ is H or linear or branched C₁-C₄ alkyl

R¹⁶ is CH₃, CH₂CH₃ or (CH₂)_qOH

q is 1 to 6

U² is N or CH;

or a dispersed polyorganosiloxane of the formula (8)



- 6 -

wherein

R^3 is as previously defined

R^{17} is OH, OR^{18} or CH_3

R^{18} is CH_3 or CH_2CH_3

R^{19} is $R^{20}-(EO)_m-(PO)_n-R^{21}$

m is 3 to 25

n is 0 to 10

R^{20} is the direct bond or $CH_2CH(R^{22})(CH_2)_pR^{23}$

p is 1 to 4

R^{21} is H, R^{24} , $CH_2CH(R^{22})NH_2$ or $CH(R^{22})CH_2NH_2$

R^{22} is H or CH_3

R^{23} is O or NH

R^{24} is linear or branched C_1 - C_8 alkyl or $Si(R^{25})_3$

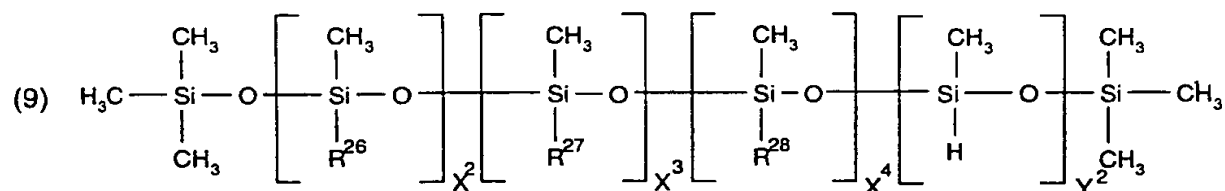
R^{25} is R^{24} , OCH_3 or OCH_2CH_3

EO is $-CH_2CH_2O-$

PO is $-CH(CH_3)CH_2O-$ or $-CH_2CH(CH_3)O-$

the sum of X_1 , Y_1 and S is 20 to 1500;

or a dispersed polyorganosiloxane of the formula (9)



wherein

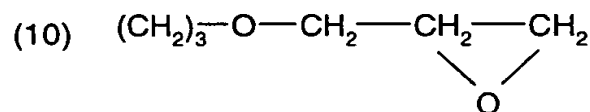
R^{26} is linear or branched C_1 - C_{20} alkoxy, $CH_2CH(R^4)R^{29}$

R^4 is as previously defined

R^{29} is linear or branched C_1 - C_{20} alkyl

R^{27} is aryl, aryl substituted by linear or branched C_1 - C_{10} alkyl, linear or branched C_1 - C_{20} alkyl substituted by aryl or aryl substituted by linear or branched C_1 - C_{10} alkyl

R^{28} is



- 7 -

the sum of X^2 , X^3 , X^4 and Y^2 is 20 to 1500, wherein X^3 , X^4 and Y^2 may be independently of each other 0;
or a mixture thereof.

The composition is preferably used as a component in a liquid rinse conditioner composition. The textile fibre materials are preferably treated for wrinkle recovery.

In tumble dryer applications the compositions are usually incorporated into impregnates on non-woven sheets. However, other application forms are known to those skilled in the art.

The fabric softener composition will be used after the textile fibre materials have been washed with a laundry detergent, which may be one of a broad range of detergent types. The tumble dryer sheet will be used after a laundering process. The textile fibre materials may be damp or dry.

The fabric softener composition may also be sprayed directly onto the fabrics prior to or during the ironing or drying of the treated fabrics.

The polyorganosiloxane may be anionic, nonionic or cationic, preferably nonionic or cationic.

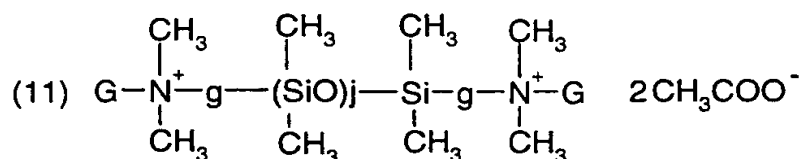
The polyorganosiloxanes, or mixtures thereof, are preferably used in a dispersed form, via the use of an emulsifier. The fabric softener compositions are preferably in liquid aqueous form. The fabric softener compositions preferably contain a water content of 25 to 90% by weight based on the total weight of the composition. The particles of the emulsion as a rule have a diameter of between 5nm and 1000nm.

When the polyorganosiloxane contains a nitrogen atom the nitrogen content of the aqueous emulsion due to the polyorganosiloxane is from 0.001 to 0.25 % with respect to the silicon content. In general a nitrogen content of 0 to 0.25% is preferred.

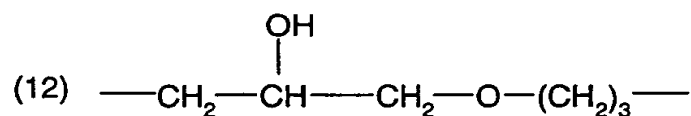
The fabric softener composition preferably has a solids content of 5 to 70% at a temperature of 120°C.

The fabric softener composition preferably has a pH value from 2.0 to 9.0, especially 2.0 to 7.0.

The fabric softener composition may further comprise an additional polyorganosiloxane:



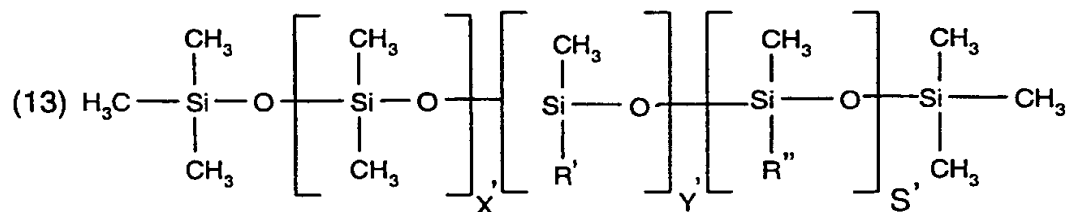
wherein g is



and G is C₁ to C₂₀ alkyl.

This polydimethylsiloxane is cationic, has a viscosity at 25°C of 250 mm²s⁻¹ to 450 mm²s⁻¹, has a specific gravity of 1.00 to 1.02 g/cm³ and has a surface tension of 28.5 mNm⁻¹ to 33.5 mNm⁻¹.

The fabric softener composition may further comprise an additional polyorganosiloxane, such as that known as Magnasoft HSSD, or a polyorganosiloxane of the formula:



R' is CH₂CH₂CH₂N(R'')₂

R'' is linear or branched C₁-C₄ alkyl

R is (CH₂)_x-(EO)_m-(PO)_n-R'''

m is 3 to 25

n is 0 to 10

X'' is 0 to 4

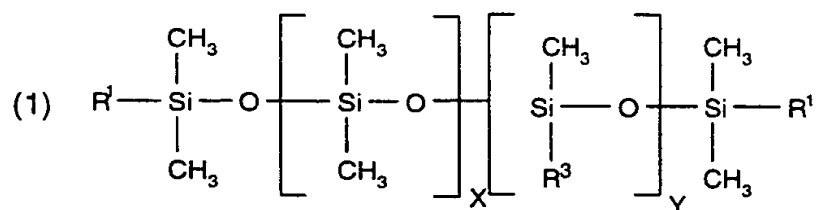
R''' is H or linear or branched C₁-C₄ alkyl

EO is -CH₂CH₂O-

PO is -CH(CH₃)CH₂O- or -CH₂CH(CH₃)O-

the sum of X', Y' and S' is 40 to 300.

Preferably the compositions comprise dispersed polyorganosiloxanes of formula (1):

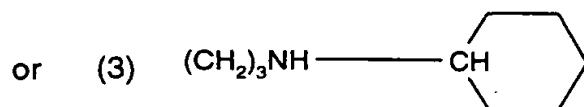
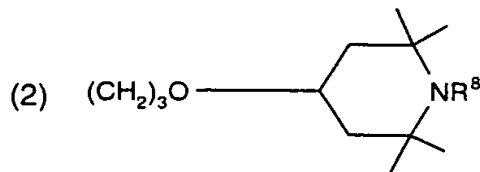


wherein

R¹ is OH, OR² or CH₃

R² is CH₃ or CH₂CH₃

R³ is C₁-C₂₀alkoxy, CH₃, CH₂CHR⁴CH₂NHR⁵, or



R⁴ is H or CH₃

R⁵ is H, CH₂CH₂NHR⁶, C(=O)-R⁷

R⁶ is H or C(=O)-R⁷

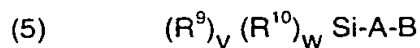
R⁷ is CH₃, CH₂CH₃ or CH₂CH₂CH₂OH

R⁸ is H or CH₃

the sum of X and Y is 40 to 1500

or a dispersed polyorganosiloxane which comprises at least one unit of the formula (5);

- 10 -



wherein

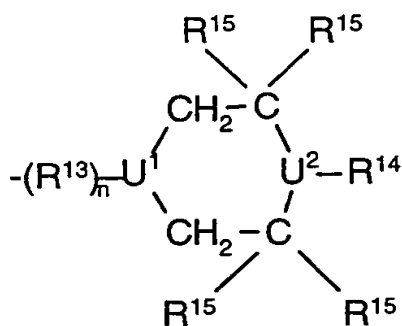
R^9 is CH_3 ,

R^{10} is $-\text{O-Si}$ or $-\text{O-R}^9$

the sum of v and w equals 3, and v does not equal 3

$A = -\text{CH}_2\text{CH}(\text{R}^{11})(\text{CH}_2)_k$

$B =$



(6)

n is 1

U^1 is CH

k is 0 to 6

R^{11} is H or CH_3

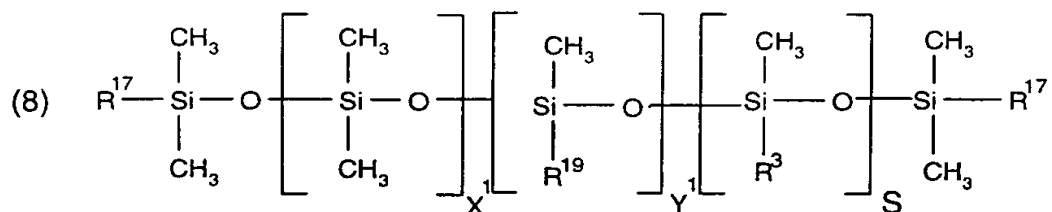
R^{13} is $\text{OOCN}(\text{Butyl})$

R^{14} is H , linear $\text{C}_1\text{-C}_4$ alkyl, Phenyl

R^{15} is H or linear $\text{C}_1\text{-C}_4$ alkyl

U^2 is N

or a dispersed polyorganosiloxane of the formula (8);



wherein

R^3 is as previously defined

R^{17} is OH, OR^{18} or CH_3

R^{18} is CH_3 or CH_2CH_3

R^{19} is $R^{20}-(EO)_m-(PO)_n-R^{21}$

m is 3 to 25

n is 0 to 10

R^{20} is the direct bond or $CH_2CH(R^{22})(CH_2)_pR^{23}$

p is 1 to 4

R^{21} is H, R^{24} , $CH_2CH(R^{22})NH_2$ or $CH(R^{22})CH_2NH_2$

R^{22} is H or CH_3

R^{23} is O or NH

R^{24} is linear or branched C_1 - C_3 alkyl or $Si(R^{25})_3$

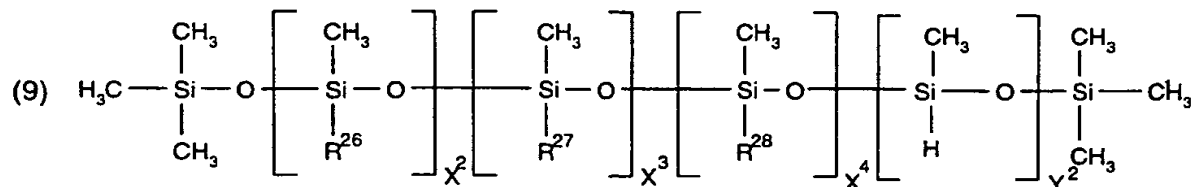
R^{25} is R^{24} , OCH_3 or OCH_2CH_3

EO is $-CH_2CH_2O-$

PO is $-CH(CH_3)CH_2O-$ or $-CH_2CH(CH_3)O-$

the sum of X_1 , Y_1 and s is 40 to 1500

or a dispersed polyorganosiloxane of the formula (9);



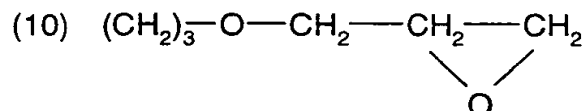
R^{26} is linear C_1 - C_{20} alkoxy, $CH_2CH(R^4)R^{29}$

R^4 is as previously defined

R^{29} is linear C_1 - C_{20} alkyl

R^{27} is, $CH_2CH(R^4)Phenyl$

- 12 -

R²⁸ is

the sum of X², X³, X⁴ and Y² is 40 to 1500, wherein X³, X⁴ and Y² may be independently of each other 0;

or a mixture thereof.

As to the polyorganosiloxanes of formula (1) the following preferences apply:

R¹ is preferably OH or CH₃.

R³ is preferably CH₃, C₁₀-C₂₀alkoxy or CH₂CHR⁴CH₂NHR⁵.

R⁴ is preferably H.

R⁵ is preferably H or CH₂CH₂NHR⁶.

R⁶ is preferably H or C(=O)-R⁷.

R⁷ is preferably CH₃, CH₂CH₃ or especially CH₂CH₂CH₂OH.

The sum of X + Y is preferably 100 to 2000.

Preferred are polyorganosiloxanes of formula (1) wherein

R¹ is OH or CH₃,

R³ is CH₃, C₁₀-C₂₀alkoxy or CH₂CHR⁴CH₂NHR⁵,

R⁴ is H,

R⁵ is H or CH₂CH₂NHR⁶,

R⁶ is H or C(=O)-R⁷, and

R⁷ is CH₃, CH₂CH₃ or especially CH₂CH₂CH₂OH.

As to the polyorganosiloxanes of formula (8) the following preferences apply:

R³ is preferably CH₃, C₁₀-C₂₀alkoxy or CH₂CHR⁴CH₂NHR⁵.

R⁴ is preferably H.

R⁵ is preferably H or CH₂CH₂NHR⁶.

R⁶ is preferably H or C(=O)-R⁷.

R⁷ is preferably CH₂CH₃, CH₂CH₂CH₂OH or especially CH₃.

R₁₇ is preferably CH₃ or OH.

R₂₀ is preferably the direct bond.

- 13 -

R₂₁ is preferably H.

Preferred are polyorganosiloxanes of formula (8) wherein

R³ is CH₃, C₁₀-C₂₀alkoxy or CH₂CHR⁴CH₂NHR⁵,

R⁴ is H,

R⁵ is H or CH₂CH₂NHR⁶,

R⁶ is H or C(=O)-R⁷,

R⁷ is CH₂CH₃, CH₂CH₂CH₂OH or especially CH₃, and

R₁₇ is CH₃ or OH.

As to the polyorganosiloxanes of formula (9) the following preferences apply:

R²⁶ is preferably CH₂CH(R⁴)R²⁹.

R⁴ is preferably H.

R²⁷ is preferably 2-phenyl propyl.

The sum of X², X³, X⁴ and Y² is preferably 40 to 500.

Preferred are polyorganosiloxanes of formula (9) wherein

R²⁶ is CH₂CH(R⁴)R²⁹,

R⁴ is H, and

R²⁷ is 2-phenyl propyl.

Preferred are polyorganosiloxanes of formulae (1), (8) and (9), especially those of formulae (1) and (8). Very interesting polyorganosiloxanes are those of formula (1).

Emulsifiers used to prepare the polyorganosiloxane compositions include:

- i) Ethoxylates, such as alkyl ethoxylates, amine ethoxylates or ethoxylated alkylammoniumhalides. Alkyl ethoxylates include alcohol ethoxylates or isotridecyl ethoxylates. Preferred alcohol ethoxylates include linear or branched nonionic alkyl ethoxylates containing 2 to 15 ethylene oxide units. Preferred isotridecyl ethoxylates include nonionic isotridecyl ethoxylates containing 5 to 25 ethylene oxide units. Preferred amine ethoxylates include nonionic C10 to C20 alkyl amino ethoxylates containing 4 to 10 ethylene oxide units. Preferred ethoxylated alkylammoniumhalides include nonionic or cationic ethoxylated C6 to C20 alkyl bis(hydroxyethyl)methylammonium chlorides.

- ii) Alkylammonium halides, preferably cationic quaternary ester alkylammonium halides.
- iii) Silicones, preferably nonionic polydimethylsiloxane polyoxyalkylene copolymers
- iv) Saccharides, preferably nonionic alkylpolyglycosides.

A mixture of these emulsifiers may also be used.

As mentioned previously, the fabric softener compositions further comprise one or more components selected from polyethylene, fatty acid alkanol amide, polysilicic acid and polyurethane. These components are described below.

The dispersed polyethylene (polyethylene wax) is known and is described in detail in the prior art (compare, for example, DE-C-2,359,966, DE-A-2,824,716 and DE-A-1,925,993). The dispersed polyethylene is as a rule a polyethylene having functional groups, in particular COOH groups, some of which can be esterified. These functional groups are introduced by oxidation of the polyethylene. However, it is also possible to obtain the functionality by copolymerization of ethylene with, for example, acrylic acid. The dispersed polyethylenes have a density of at least 0.91 g/cm^3 at 20°C ., an acid number of at least 5 and a saponification number of at least 10. It is preferred that the drop point is in the range of from 100 to 150°C . Dispersed polyethylenes which have a density of 0.95 to 1.05 g/cm^3 at 20°C ., an acid number of 10 to 60 and a saponification number of 15 to 80 are particularly preferred. This material is generally obtainable commercially in the form of flakes, lozenges and the like. A mixture of these dispersed polyethylenes may also be used.

The polyethylene wax is usually employed in the form of dispersions. Various emulsifiers are suitable for their preparation. The preparation of the dispersions is described in detail in the prior art.

Emulsifiers suitable for dispersing the polyethylene component include:

- i) Ethoxylates, such as alkyl ethoxylates or amine ethoxylates. Alkyl ethoxylates include alcohol ethoxylates or isotridecyl ethoxylates. Preferred alcohol ethoxylates include nonionic fatty alcohol ethoxylates containing 2 to 55 ethylene oxide units. Preferred isotridecyl ethoxylates include nonionic isotridecyl ethoxylates containing 6 to 9 ethylene

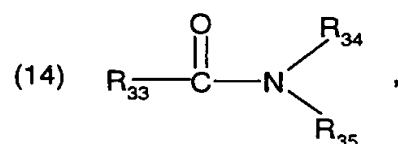
- 15 -

oxide units. Preferred amine ethoxylates include nonionic C10 to C20 alkyl amino ethoxylates containing 7 to 9 ethylene oxide units.

- ii) Alkylammonium halides, preferably cationic quaternary ester alkylammonium halides.
- iii) Ammonium salts, preferably cationic aliphatic quaternary ammonium chloride or sulfate.

A mixture of these emulsifiers may also be used.

Suitable fatty acid alkanolamides are for example those of formula



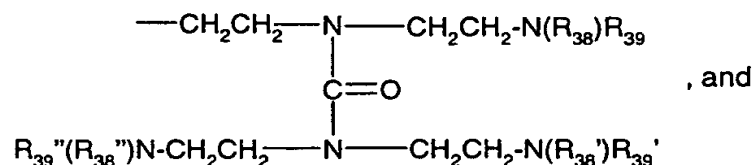
wherein

R_{33} is a saturated or unsaturated hydrocarbon radical containing 10 to 24 carbon atoms,

R_{34} is hydrogen or a radical of formula $-\text{CH}_2\text{OH}$, $-(\text{CH}_2\text{CH}_2\text{O})_c\text{H}$ or $-\overset{\text{O}}{\parallel}{\text{C}}-\text{R}_{36}$ wherein c is a

number from 1 to 10 and R_{36} is as defined above for R_{33} , and

R_{35} is a radical of formula $-\text{CH}_2\text{OH}$, $-(\text{CH}_2\text{CH}_2\text{O})_c\text{H}$, $-\text{CH}_2\text{CH}_2-\text{N} \begin{array}{l} (\text{CH}_2\text{CH}_2\text{O})_c\text{H} \\ \text{R}_{37} \end{array}$ or

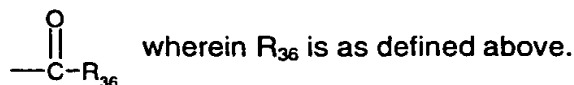


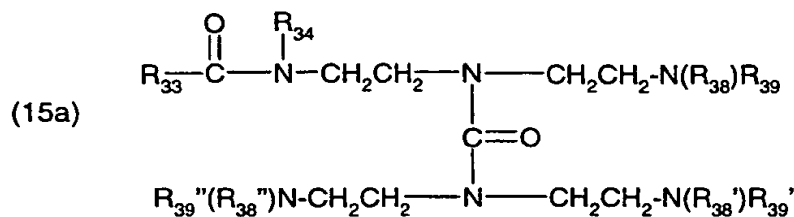
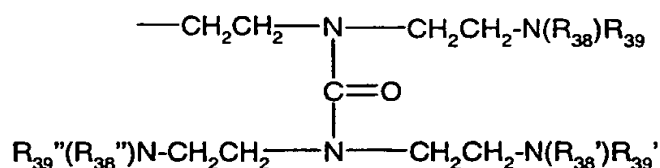
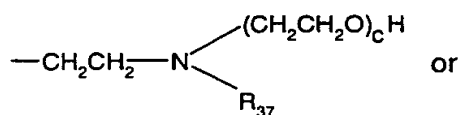
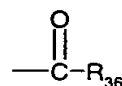
c is as defined above,

R_{37} is hydrogen or a radical of formula $-\overset{\text{O}}{\parallel}{\text{C}}-\text{R}_{36}$ wherein R_{36} is as defined above,

R_{38} , R_{38}' and R_{38}'' have the same or different meaning and are as defined above for R_{34} , and

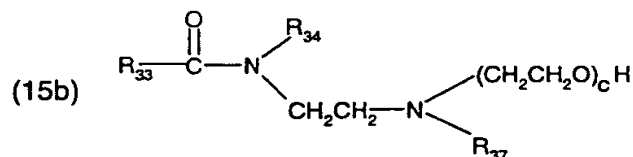
R_{39} , R_{39}' and R_{39}'' have the same or different meaning and are a radical of formula





R_{34} , R_{38} , R_{38}' and R_{38}'' are hydrogen or $-\text{CH}_2\text{OH}$.

Furthermore, fatty acid alkanolamides of formula



are preferred, wherein R_{33} , R_{34} , R_{37} and c are as defined above.

Preferred are fatty acid alkanolamides of formula (15b), wherein

R_{34} and R_{37} are hydrogen or a radical of formula $\begin{array}{c} \text{O} \\ \parallel \\ -\text{C}-\text{R}_{36} \end{array}$. R_{34} is preferably hydrogen.

The above fatty acid alkanolamides can also be present in form of the corresponding ammonium salts.

A mixture of these fatty acid alkanolamides may also be used.

Emulsifiers suitable for dispersing the fatty acid alkanol amide component include:

- i) Ethoxylates, such as alkyl ethoxylates, amine ethoxylates or amide ethoxylates. Alkyl ethoxylates include alcohol ethoxylates or isotridecyl ethoxylates. Preferred alcohol ethoxylates include nonionic fatty alcohol ethoxylates containing 2 to 55 ethylene oxide units. Preferred isotridecyl ethoxylates include nonionic isotridecyl ethoxylates containing 5 to 45 ethylene oxide units. Preferred amine ethoxylates include nonionic C10 to C20 alkyl amino ethoxylates containing 4 to 25 ethylene oxide units. Preferred amide ethoxylates include cationic fatty acid amide ethoxylates containing 2 to 25 ethylene oxide units.
- ii) Alkylammonium halides, preferably cationic quaternary ester alkylammonium halides or cationic aliphatic acid alkylamidotrialkylammonium methosulfates.
- iii) Ammonium salts, preferably cationic aliphatic quaternary ammonium chloride or sulfate.

A mixture of these emulsifiers may also be used.

Examples for polyurethanes are the reaction products of a diol and an ethoxysilicate with a diisocyanate.

The additives selected from the group consisting of a polyethylene, a fatty acid alkanolamide, a polysilicic acid, and a polyurethane are, as a rule, used in an amount of 0.01 to 25 % by weight, especially 0.01 to 15 % by weight, based on the total weight of the fabric softener composition. An amount of 0.05 to 15 % by weight, especially 0.1 to 15 % by weight, is preferred. Highly preferred is an upper limit of 10 %, especially 5 %.

Preferred as additives are polyethylene, fatty acid alkanolamides and polyurethanes, especially polyethylene and fatty acid alkanolamides. Highly preferred are polyethylene.

A highly preferred fabric softener composition used according to the present invention comprises:

- a) 0.01 to 70 % by weight based on the total weight of the composition of a polyorganosiloxane, or a mixture thereof;
- b) 0.2 to 25 % by weight based on the total weight of an emulsifier, or a mixture thereof;
- c) 0.01 to 25 % by weight, especially 0.01 to 15 % by weight, based on the total weight of at least one additive selected from the group consisting of a polyethylene, a fatty acid alkanolamide, a polysilicic acid, or a polyurethane, and
- d) water to 100 %.

The fabric softener compositions can be prepared as follows:

Firstly, emulsions of the polyorganosiloxane are prepared. The polyorganosiloxane and polyethylene, fatty acid alkanol amide, polysilicic acid or polyurethane are emulsified in water using one or more surfactants and shear forces, e.g. by means of a colloid mill. Suitable surfactants are described above. The components may be emulsified individually before being mixed together, or emulsified together after the components have been mixed. The surfactant(s) is/are used in customary amounts known to the person skilled in the art and can be added either to the polyorganosiloxane or to the water prior to emulsification. Where appropriate, the emulsification operation can be carried out at elevated temperature. The fabric softener composition according to the invention is usually, but not exclusively,

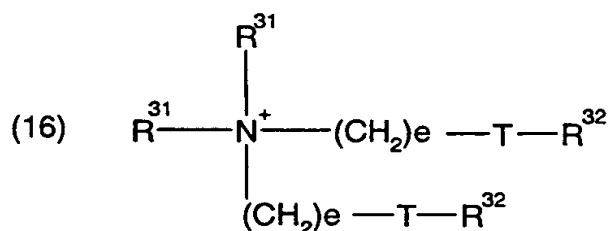
prepared by firstly stirring the active substance, i.e. the hydrocarbon based fabric softening component, in the molten state into water, then, where required, adding further desired additives and, finally, after cooling, adding the polyorganosiloxane emulsion.

Hydrocarbon fabric softeners suitable for use herein are selected from the following classes of compounds:

(i) Cationic quaternary ammonium salts. The counter ion of such cationic quaternary ammonium salts may be a halide, such as chloride or bromide, methyl sulphate, or other ions well known in the literature. Preferably the counter ion is methyl sulfate or any alkyl sulfate or any halide, methyl sulfate being most preferred for the dryer-added articles of the invention.

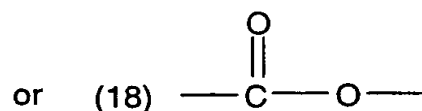
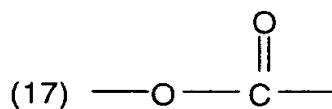
Examples of cationic quaternary ammonium salts include but are not limited to:

(1) Acyclic quaternary ammonium salts having at least two C_8 to C_{30} , preferably C_{12} to C_{22} alkyl or alkenyl chains, such as: ditallowdimethyl ammonium methylsulfate, di(hydrogenated tallow)dimethyl ammonium methylsulfate, distearyldimethyl ammonium methylsulfate, dicocodimethyl ammonium methylsulfate and the like. It is especially preferred if the fabric softening compound is a water insoluble quaternary ammonium material which comprises a compound having two C_{12} to C_{18} alkyl or alkenyl groups connected to the molecule via at least one ester link. It is more preferred if the quaternary ammonium material has two ester links present. An especially preferred ester-linked quaternary ammonium material for use in the invention can be represented by the formula:



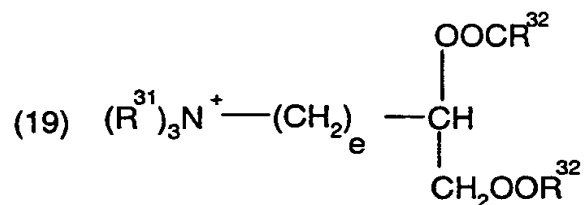
wherein each R^{31} group is independently selected from C_1 to C_4 alkyl, hydroxyalkyl or C_2 to C_4 alkenyl groups; T is either

- 20 -



and wherein each R^{32} group is independently selected from C_8 to C_{28} alkyl or alkenyl groups; and e is an integer from 0 to 5.

A second preferred type of quaternary ammonium material can be represented by the formula:



wherein R^{31} , e and R^{32} are as defined above.

(2) Cyclic quaternary ammonium salts of the imidazolinium type such as di(hydrogenated tallow)dimethyl imidazolinium methylsulfate, 1-ethylene-bis(2-tallow-1-methyl) imidazolinium methylsulfate and the like;

(3) Diamido quaternary ammonium salts such as: methyl-bis(hydrogenated tallow amidoethyl)-2-hydroxyethyl ammonium methyl sulfate, methyl bi(tallowamidoethyl)-2-hydroxypropyl ammonium methylsulfate and the like;

(4) Biodegradable quaternary ammonium salts such as N,N-di(tallowoyl-oxy-ethyl)-N,N-dimethyl ammonium methyl sulfate and N,N-di(tallowoyl-oxy-propyl)-N,N-dimethyl ammonium methyl sulfate. Biodegradable quaternary ammonium salts are described, for example, in U.S. Patents 4,137,180, 4,767,547 and 4,789,491 incorporated by reference herein.

Preferred biodegradable quaternary ammonium salts include the biodegradable cationic diester compounds as described in U.S. Patent 4,137,180, herein incorporated by reference.

(ii) Tertiary fatty amines having at least one and preferably two C8 to C30, preferably C12 to C22 alkyl chains. Examples include hardened tallow-di-methylamine and cyclic amines such as 1-(hydrogenated tallow)amidoethyl-2-(hydrogenated tallow) imidazoline. Cyclic amines which may be employed for the compositions herein are described in U.S. Patent 4,806,255 incorporated by reference herein.

(iii) Carboxylic acids having 8 to 30 carbon atoms and one carboxylic group per molecule. The alkyl portion has 8 to 30, preferably 12 to 22 carbon atoms. The alkyl portion may be linear or branched, saturated or unsaturated, with linear saturated alkyl preferred. Stearic acid is a preferred fatty acid for use in the composition herein. Examples of these carboxylic acids are commercial grades of stearic acid and palmitic acid, and mixtures thereof which may contain small amounts of other acids.

(iv) Esters of polyhydric alcohols such as sorbitan esters or glycerol stearate. Sorbitan esters are the condensation products of sorbitol or iso-sorbitol with fatty acids such as stearic acid. Preferred sorbitan esters are monoalkyl. A common example of sorbitan ester is SPAN 60 (ICI) which is a mixture of sorbitan and isosorbide stearates.

(v) Fatty alcohols, ethoxylated fatty alcohols, alkyphenols, ethoxylated alkyphenols, ethoxylated fatty amines, ethoxylated monoglycerides and ethoxylated diglycerides.

(vi) Mineral oils, and polyols such as polyethylene glycol.

These softeners are more definitively described in U.S. Patent 4,134,838 the disclosure of which is incorporated by reference herein. Preferred fabric softeners for use herein are acyclic quaternary ammonium salts. Di(hydrogenated)tallowdimethyl ammonium methylsulfate is most widely used for dryer articles of this invention. Mixtures of the above mentioned fabric softeners may also be used.

- 22 -

The fabric softening composition employed in the present invention preferably contains about 0.1% to about 95% of the fabric softening component. Preferably from about 2% to about 70% and most preferably from about 2% to about 30% of the fabric softening component is employed herein to obtain optimum softening at minimum cost. When the fabric softening component includes a quaternary ammonium salt, the salt is used in the amount of about 2% to about 70%, preferably about 2% to about 30%.

The fabric softener composition can, for example, be prepared by mixing a preformulated fabric softener with an emulsion comprising the polyorganosiloxane and the additive.

The liquid rinse conditioner composition may also comprise additives which are customary for standard commercial liquid rinse conditioners, for example alcohols, such as ethanol, n-propanol, i-propanol, polyhydric alcohols, for example glycerol and propylene glycol; amphoteric and nonionic surfactants, for example carboxyl derivatives of imidazole, oxyethylated fatty alcohols, hydrogenated and ethoxylated castor oil, alkyl polyglycosides, for example decyl polyglucose and dodecylpolyglucose, fatty alcohols, fatty acid esters, fatty acids, ethoxylated fatty acid glycerides or fatty acid partial glycerides; also inorganic or organic salts, for example water-soluble potassium, sodium or magnesium salts, non-aqueous solvents, pH buffers, perfumes, dyes, hydrotropic agents, antifoams, anti redeposition agents, polymeric or other thickeners, enzymes, optical brighteners, antishrink agents, stain removers, germicides, fungicides, antioxidants and corrosion inhibitors.

These fabric softener compositions are traditionally prepared as dispersions containing for example up to 20 % by weight of active material in water. They have a turbid appearance. However, alternative formulations usually containing actives at levels of 5 to 40 % along with solvents can be prepared as microemulsions which have a clear appearance (as to the solvents and the formulations see for example US-A-5,543,067 und WO-A-98/17757). The additives and polyorganosiloxanes of the present invention can be used for such compositions although it will be necessary to use them in microemulsion form to preserve the clear appearance of the fabric softener compositions which are microemulsions.

Another aspect of the invention is a tumble dryer sheet article. The conditioning composition of the present invention may be coated onto a flexible substrate which carries a fabric conditioning amount of the composition and is capable of releasing the composition at dryer

- 23 -

operating temperatures. The conditioning composition in turn has a preferred melting (or softening) point of about 25°C to about 150°C.

The fabric conditioning composition which may be employed in the invention is coated onto a dispensing means which effectively releases the fabric conditioning composition in a tumble dryer. Such dispensing means can be designed for single usage or for multiple uses. One such multi-use article comprises a sponge material releasably enclosing enough of the conditioning composition to effectively impart fabric softness during several drying cycles. This multi-use article can be made by filling a porous sponge with the composition. In use, the composition melts and leaches out through the pores of the sponge to soften and condition fabrics. Such a filled sponge can be used to treat several loads of fabrics in conventional dryers, and has the advantage that it can remain in the dryer after use and is not likely to be misplaced or lost.

Another article comprises a cloth or paper bag releasably enclosing the composition and sealed with a hardened plug of the mixture. The action and heat of the dryer opens the bag and releases the composition to perform its softening.

A highly preferred article comprises the inventive compositions releasably affixed to a flexible substrate such as a sheet of paper or woven or non-woven cloth substrate. When such an article is placed in an automatic laundry dryer, the heat, moisture, distribution forces and tumbling action of the dryer removes the composition from the substrate and deposits it on the fabrics.

The sheet conformation has several advantages. For example, effective amounts of the compositions for use in conventional dryers can be easily absorbed onto and into the sheet substrate by a simple dipping or padding process. Thus, the end user need not measure the amount of the composition necessary to obtain fabric softness and other benefits.

Additionally, the flat configuration of the sheet provides a large surface area which results in efficient release and distribution of the materials onto fabrics by the tumbling action of the dryer.

The substrates used in the articles can have a dense, or more preferably, open or porous structure. Examples of suitable materials which can be used as substrates herein include

- 24 -

paper, woven cloth, and non-woven cloth. The term "cloth" herein means a woven or non-woven substrate for the articles of manufacture, as distinguished from the term "fabric" which encompasses the clothing fabrics being dried in an automatic dryer.

It is known that most substances are able to absorb a liquid substance to some degree; however, the term "absorbent", as used herein, is intended to mean a substrate with an absorbent capacity (i.e., a parameter representing a substrate's ability to take up and retain a liquid) from 4 to 12, preferably 5 to 7 times its weight of water.

If the substrate is a foamed plastics material, the absorbent capacity is preferably in the range of 15 to 22, but some special foams can have an absorbent capacity in the range from 4 to 12.

Determination of absorbent capacity values is made by using the capacity testing procedures described in U.S. Federal Specifications (UU-T-595b), modified as follows:

1. tap water is used instead of distilled water;
2. the specimen is immersed for 30 seconds instead of 3 minutes;
3. draining time is 15 seconds instead of 1 minute; and
4. the specimen is immediately weighed on a torsion balance having a pan with turned-up edges.

Absorbent capacity values are then calculated in accordance with the formula given in said Specification. Based on this test, one-ply, dense bleached paper (e.g., Kraft or bond having a basis weight of about 32 pounds per 3,000 square feet) has an absorbent capacity of 3.5 to 4; commercially available household one-ply towel paper has a value of 5 to 6; and commercially available two-ply household towelling paper has a value of 7 to about 9.5.

Suitable materials which can be used as a substrate in the invention herein include, among others, sponges, paper, and woven and non-woven cloth, all having the necessary absorbency requirements defined above.

The preferred non-woven cloth substrates can generally be defined as adhesively bonded fibrous or filamentous products having a web or carded fiber structure (where the fiber strength is suitable to allow carding), or comprising fibrous mats in which the fibers or

- 25 -

filaments are distributed haphazardly or in random array (i.e. an array of fibers is a carded web wherein partial orientation of the fibers is frequently present, as well as a completely haphazard distributional orientation), or substantially aligned. The fibers or filaments can be natural (e.g. wool, silk, jute, hemp, cotton, linen, sisal, or ramie) or synthetic (e.g. rayon, cellulose ester, polyvinyl derivatives, polyolefins, polyamides, or polyesters).

The preferred absorbent properties are particularly easy to obtain with non-woven cloths and are provided merely by building up the thickness of the cloth, i.e., by superimposing a plurality of carded webs or mats to a thickness adequate to obtain the necessary absorbent properties, or by allowing a sufficient thickness of the fibers to deposit on the screen. Any diameter or denier of the fiber (generally up to about 10 denier) can be used, inasmuch as it is the free space between each fiber that makes the thickness of the cloth directly related to the absorbent capacity of the cloth, and which, further, makes the non-woven cloth especially suitable for impregnation with a composition by means of intersectional or capillary action. Thus, any thickness necessary to obtain the required absorbent capacity can be used.

When the substrate for the composition is a non-woven cloth made from fibers deposited haphazardly or in random array on the screen, the articles exhibit excellent strength in all directions and are not prone to tear or separate when used in the automatic clothes dryer.

Preferably, the non-woven cloth is water-laid or air-laid and is made from cellulosic fibers, particularly from regenerated cellulose or rayon. Such non-woven cloth can be lubricated with any standard textile lubricant.

Preferably, the fibers are from 5mm to 50mm in length and are from 1.5 to 5 denier. Preferably, the fibers are at least partially orientated haphazardly, and are adhesively bonded together with a hydrophobic or substantially hydrophobic binder-resin. Preferably, the cloth comprises about 70% fiber and 30% binder resin polymer by weight and has a basis weight of from about 18 to 45g per square meter.

In applying the fabric conditioning composition to the absorbent substrate, the amount impregnated into and/or coated onto the absorbent substrate is conveniently in the weight ratio range of from about 10:1 to 0.5:1 based on the ratio of total conditioning composition to

dry, untreated substrate (fiber plus binder). Preferably, the amount of the conditioning composition ranges from about 5:1 to about 1:1, most preferably from about 3:1 to 1:1, by weight of the dry untreated substrate.

According to one preferred embodiment of the invention, the dryer sheet substrate is coated by being passed over a rotogravure applicator roll. In its passage over this roll, the sheet is coated with a thin, uniform layer of molten fabric softening composition contained in a rectangular pan at a level of about 15g per square yard. Passage for the substrate over a cooling roll then solidifies the molten softening composition to a solid. This type of applicator is used to obtain a uniform homogeneous coating across the sheet.

Following application of the liquefied composition, the articles are held at room temperature until the composition substantially solidifies. The resulting dry articles, prepared at the composition substrate ratios set forth above, remain flexible; the sheet articles are suitable for packaging in rolls. The sheet articles can optionally be slitted or punched to provide a non-blocking aspect at any convenient time if desired during the manufacturing process.

The fabric conditioning composition employed in the present invention includes certain fabric softeners which can be used singly or in admixture with each other.

Examples of suitable textile fibre materials which can be treated with the liquid rinse conditioner composition are materials made of silk, wool, polyamide, acrylics or polyurethanes, and, in particular, cellulosic fibre materials of all types. Such fibre materials are, for example, natural cellulose fibres, such as cotton, linen, jute and hemp, and regenerated cellulose. Preference is given to textile fibre materials made of cotton. The fabric softener compositions are also suitable for hydroxyl-containing fibres which are present in mixed fabrics, for example mixtures of cotton with polyester fibres or polyamide fibres.

A better understanding of the present invention and of its many advantages will be had by referring to the following Examples, given by way of illustration. The percentages given in the examples are percentages by weight.

Example 1 (preparation of the rinse conditioners)

The liquid rinse conditioners are prepared by using the procedure described below. This type of fabric rinse conditioners is normally known under the name of "triple strength" or "triple fold" formula.

75 % by weight of the total amount of water is heated to 40°C. The molten fabric softener di-(palmcarboxyethyl)-hydroxyethyl-methylammonium-methosulfate (or Rewoquat WE 38 DPG available from Witco) is added to the heated water under stirring and the mixture is stirred for 1 hour at 40°C. Afterwards the aqueous softener solution is cooled down to below 30°C while stirring. When the solution cools down sufficiently magnesium chloride is added and the pH is adjusted to 3.2 with 0.1 N hydrochloric acid. The formulation is then filled up with water to 100%.

The rinse conditioner formulation as described above was used as a base formulation. In a final step the fabric softener is mixed with a separately prepared polyorganosiloxane /additive emulsion. The fabric softener formulations used in the following examples are listed in the following Table 1.

Table 1 (rinse conditioner formulations used in the application test for 1 kg wash load)

Rinse conditioner formulation	Polyorgano-siloxane emulsion (calculated on solid content of the emulsion)	Fabric softener Base Formulation	pH
0 (Reference)	-----	13.3 g	3.2
A	0.2 g of Type I	13.3 g	3.2
B	0.2 g of Type II	13.3 g	3.2
C	0.2 g of Type III	13.3 g	3.2
D	0.2 g of Type IV	13.3 g	3.2
E	0.2 g of Type V	13.3 g	3.2
F	0.2 g of Type VI	13.3 g	3.2
G	0.2 g of Type VII	13.3 g	3.2
H	0.2 g of Type VIII	13.3 g	3.2
I	0.2 g of Type IX	13.3 g	3.2

J	0.2 g of Type X	13.3 g	3.2
K	0.2 g of Type XI	13.3 g	3.2
L	0.2 g of Type XII	13.3 g	3.2
M	0.2 g of Type XIII	13.3 g	3.2
N	0.2 g of Type XIV	13.3 g	3.2
O	0.2 g of Type XV	13.3 g	3.2
P	0.2 g of Type XVI	13.3 g	3.2
Q	0.2 g of Type XVII	13.3 g	3.2
R	0.2 g of Type XXII	13.3 g	3.2
S	0.2 g of Type XXIII	13.3 g	3.2

Types of polyorganosiloxane emulsions used

Type I

- Polyorganosiloxane of general formula (1), wherein R_1 is -OH, R_3 is -CH₃,
 $X + Y = 300-1500$, % nitrogen (with respect to silicone) = 0
- 3.7% of an emulsifier
- 12.5% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm³ at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80
- solid content of the emulsion measured by evaporation at 120°C = 27.0-29.0%
- water content = 71.3%

Type II

- Polyorganosiloxane of general formula (1), wherein R_1 is -OH, R_3 is -CH₃,
 $X + Y = 300-1500$, % nitrogen (with respect to silicone) = 0
- 4.1% of an emulsifier
- 7.8% of a fatty acid dialkanolamide of formula (15a), wherein R_{34} , R_{38} , R_{38}' and R_{38}'' are hydrogen or -CH₂OH
- solid content of the emulsion measured by evaporation at 120°C = 23.5-25.5%
- water content = 75%

Type III

- Polyorganosiloxane of general formula (1), wherein R_1 is $-OH$, R_3 is $-CH_2CH_2CH_2NH_2$, $X + Y = 300-1500$,
% nitrogen (with respect to silicone) = 0.025
- 4.5 % of an emulsifier
- 1% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm^3 at 20°C , a drop point of $100-150^\circ\text{C}$, an acid number of 10 to 60 and a saponification number of 15 to 80
- solid content of the emulsion measured by evaporation at $120^\circ\text{C} = 37.0-39.0\%$
- water content = 60.7%

Type IV

- Polyorganosiloxane of general formula (1), wherein R_1 is $-CH_3$, R_3 is $-CH_2CH_2CH_2NH_2$, $X + Y = 150-300$,
% nitrogen (with respect to silicone) = 0.07
- 11% of an emulsifier
- 0.65% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm^3 at 20°C , a drop point of $100-150^\circ\text{C}$, an acid number of 10 to 60 and a saponification number of 15 to 80
- solid content of the emulsion measured by evaporation at $120^\circ\text{C} = 27.0-30.0\%$
- water content = 60.7%

Type V

- Polyorganosiloxane of general formula (1), wherein R_1 is $-OH$, R_3 is $-CH_2CH_2CH_2N(H)(CH_2CH_2NH_2)$, $X + Y = 300-1500$,
% nitrogen (with respect to silicone) = 0.03
- 3.6% of an emulsifier
- 14% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm^3 at 20°C , a drop point of $100-150^\circ\text{C}$, an acid number of 10 to 60 and a saponification number of 15 to 80
- solid content of the emulsion measured by evaporation at $120^\circ\text{C} = 23.0-25.0\%$
- water content = 73.7%

- 30 -

Type VI

- Polyorganosiloxane of general formula (1), wherein R_1 is $-\text{OH}$, R_3 is $-\text{CH}_2\text{CH}_2\text{CH}_2\text{N}(\text{H})(\text{CH}_2\text{CH}_2\text{NH}_2)$, $X + Y = 300-1500$,
% nitrogen (with respect to silicone) = 0.11
- 4.4% of an emulsifier
- 0.2% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm^3 at 20°C , a drop point of $100-150^\circ\text{C}$, an acid number of 10 to 60 and a saponification number of 15 to 80
- solid content of the emulsion measured by evaporation at $120^\circ\text{C} = 37.0-39.0\%$
- water content = 60.7%

Type VII

- Polyorganosiloxane of general formula (1), wherein R_1 is $-\text{CH}_3$, R_3 is $-\text{CH}_2\text{CH}_2\text{CH}_2\text{N}(\text{H})(\text{CH}_2\text{CH}_2\text{NH}_2)$, $X + Y = 150-300$,
% nitrogen (with respect to silicone) = 0.09
- 6.8% of an emulsifier
- 0.1% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm^3 at 20°C , a drop point of $100-150^\circ\text{C}$, an acid number of 10 to 60 and a saponification number of 15 to 80
- solid content of the emulsion measured by evaporation at $120^\circ\text{C} = 16.5-18.5\%$
- water content = 80.4%

Type VIII

- Polyorganosiloxane of general formula (1), wherein R_1 is $-\text{CH}_3$, R_3 is $-\text{CH}_2\text{CH}_2\text{CH}_2\text{N}(\text{H})(\text{CH}_2\text{CH}_2\text{NH}_2)$, $X + Y = 150-300$,
% nitrogen (with respect to silicone) = 0.12
- 11.0% of an emulsifier
- 0.3% of a fatty acid dialkanolamide of formula (15a), wherein R_{34} , R_{38} , R_{38}' and R_{38}'' are hydrogen or $-\text{CH}_2\text{OH}$
- solid content of the emulsion measured by evaporation at $120^\circ\text{C} = 24.0-26.0\%$
- water content = 72.1%

Type IX

- Polyorganosiloxane of general formula (1), wherein R_1 is $-\text{CH}_3$,

- 31 -

R_3 is $-\text{CH}_2\text{CH}_2\text{CH}_2\text{N}(\text{H})(\text{CH}_2\text{CH}_2\text{NH}_2)$, $X + Y = 40-150$,

% nitrogen (with respect to silicone) = 0.08

- 13.2% of an emulsifier

- 0.23% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm³ at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80

- solid content of the emulsion measured by evaporation at 120°C = 41.0-43.0%

- water content = 44.4%

Type X

- Polyorganosiloxane of general formula (1), wherein R_1 is $-\text{CH}_3$,

R_3 is $-\text{CH}_2\text{CH}_2\text{CH}_2\text{N}(\text{H})(\text{CH}_2\text{CH}_2\text{N}(\text{H})((\text{CO})(\text{CH}_2\text{CH}_2\text{CH}_2\text{OH})))$, $X + Y = 300-1500$,

% nitrogen (with respect to silicone) = 0.1

- 9.8% of an emulsifier

- 0.1% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm³ at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80

- solid content of the emulsion measured by evaporation at 120°C = 20.5-22.5%

- water content = 76.9%

Type XI

- Polyorganosiloxane of general formula (8), wherein R_{17} is $-\text{CH}_3$, R_3 is $-\text{CH}_3$,

R_{19} is a polyethylenoxide radical, $X^1 + Y^1 + S = 40-150$,

% nitrogen (with respect to silicone) = 0

- 2% of an emulsifier

- 0.15% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm³ at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80

- solid content of the emulsion measured by evaporation at 120°C = 23.0-25.0%

- water content = 74.9%

Type XII

- Polyorganosiloxane of general formula (8), wherein R_{17} is $-\text{CH}_3$,

R_3 is $-\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2$, R_{19} is a polyethylene/polypropyleneoxide radical,

- 32 -

$$X^1 + Y^1 + S = 150-300$$

% nitrogen (with respect to silicone) = 0.044

- 2.5% of an emulsifier
- 2.94% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm³ at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80
- solid content of the emulsion measured by evaporation at 120°C = 15.5-17.5%
- water content = 80.4%

Type XIII

- Polyorganosiloxane of general formula (8), wherein R₁₇ is -CH₃,
R₃ is -CH₂CH₂CH₂NH₂, R₁₉ is a polyethylene/polypropyleneoxide radical,

$$X^1 + Y^1 + S = 150-300$$

% nitrogen (with respect to silicone) = 0.07

- 3.5% of an emulsifier
- 1.5% of a fatty acid dialkanolamide of formula (15a), wherein R₃₄, R₃₈, R₃₈' and R₃₈'' are hydrogen or -CH₂OH
- solid content of the emulsion measured by evaporation at 120°C = 19.5-21.5%
- water content = 73%

Type XIV

- Polyorganosiloxane of general formula (8), wherein R₁₇ is -CH₃/-OH,
R₃ is -CH₂CH₂CH₂NH₂, R₁₉ is a polyethylene/polypropyleneoxide radical,

$$X^1 + Y^1 + S = 300-1500$$

% nitrogen (with respect to silicone) = 0.03

- 7.2% of an emulsifier
- 1.23% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm³ at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80
- solid content of the emulsion measured by evaporation at 120°C = 14.0-16.0%
- water content = 82.8%

Type XV

- Polyorganosiloxane of general formula (8), where in R₁₇ is -CH₃,

- 33 -

R_3 is $-\text{CH}_2\text{CH}_2\text{CH}_2\text{N}(\text{H})((\text{CH}_2\text{CH}_2\text{N}(\text{H})(\text{COCH}_3))$, R_{19} is a polyethylene/polypropyleneoxide radical, $X^1 + Y^1 + S = 150-300$

% nitrogen (with respect to silicone) = 0.015

- 7% of an emulsifier

- 9.2% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm³ at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80

- solid content of the emulsion measured by evaporation at 120°C = 18-20%

- water content = 77%

Type XVI

- Polyorganosiloxane of general formula (9), wherein R_{26} is C_{12} alkyl, R_{27} is 2-phenylpropyl, R_{28} is an epoxy radical of formula (10), $X^2 + X^3 + X^4 + Y^2 = 40-150$,

% nitrogen (with respect to silicone) = 0

- 2.9% of an emulsifier

- 0.85% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm³ at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80

- solid content of the emulsion measured by evaporation at 120°C = 37.0-39.0%

- water content = 62%

Type XVII

- Polyorganosiloxane of general formula (1), wherein R_1 is $-\text{CH}_3$, R_3 is C_{18} alkoxy,

$X+Y = 40-150$, % nitrogen (with respect to silicone) = 0

- 3.2% of an emulsifier

- 1.5% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm³ at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80

- solid content of the emulsion measured by evaporation at 120°C = 34.0-35.5%

- water content = 61.4%

Type XVIII

- Polyorganosiloxane of general formula (8), wherein R_{17} is $-\text{CH}_3$,

R_3 is $-\text{CH}_3$, R_{19} is a polyethylene/polypropyleneoxide radical,

- 34 -

$$X^1 + Y^1 + S = 150-300$$

% nitrogen (with respect to silicone) = 0

- 3% of an emulsifier

- 0.15% of an emulsifiable oxidised polyethylene which has a density of 0.95 to 1.05 g/cm³ at 20°C, a drop point of 100-150°C, an acid number of 10 to 60 and a saponification number of 15 to 80

- solid content of the emulsion measured by evaporation at 120°C = 30-32%

- water content = 63.9%.

Type XIX

- Polyorganosiloxane of general formula (11), $j = 300$,

% nitrogen (with respect to silicone) = 0.04-0.06

- 9% of an emulsifier

- solid content of the emulsion measured by evaporation at 120°C = 21-23%

- water content = 73%

Type XX

- Polyorganosiloxane of general formula (1), wherein R_1 is -OH,

R_3 is $-\text{CH}_2\text{CH}_2\text{CH}_2\text{N}(\text{H})(\text{CH}_2\text{CH}_2\text{NH}_2)$, $X + Y = 300-1500$,

% nitrogen (with respect to silicone) = 0.1

- 4.2% of an emulsifier

- 6.2% of a fatty acid monoalkanolamide of formula (15b), wherein R_{34} is hydrogen and R_{37} is hydrogen or a radical of formula $-\text{C}(\text{O})\text{R}_{36}$

- solid content of the emulsion measured by evaporation at 120°C = 38-40%

- water content = 60%

Type XXI

- Polyorganosiloxane of general formula (8), wherein R_{17} is $-\text{CH}_3$, R_3 is $-\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2$,

R_{19} is a polyethylenoxide radical, $X^1 + Y^1 + S = 40-150$,

% nitrogen (with respect to silicone) = 0.04

- 7.2% of an emulsifier

- 6.2% of a fatty acid monoalkanolamide of formula (15b), wherein R_{34} is hydrogen and R_{37} is hydrogen or a radical of formula $-\text{C}(\text{O})\text{R}_{36}$

- solid content of the emulsion measured by evaporation at 120°C = 54-56%

- water content = 38.1%

Type XXII

Mixture of 1 part of emulsion Type XX and 9 parts of emulsion Type XXI.

Type XXIII

Mixture of 1 part of emulsion Type XVIII and 1 part of emulsion Type XIX.

Example 2 (Reduction of micro creases on cotton (prior to ironing))

The formulated rinse conditioners (see Table 1) are applied according to the following procedure:

Woven cotton swatches of size of 50 cm by 40 cm are washed together with ballast material (cotton and cotton/polyester) in a AEG Oeko Lavamat 73729 washing machine maintaining the washing temperature at 40°C. The total fabric load of 1 kg is washed for 15 minutes with 33 g of ECE Color Fastness Test Detergent 77 (Formulation January 1977, according to ISO 105-CO6). The rinse conditioner formulation as described in Table 1 is applied in the last rinse cycle at 20°C. After rinsing with the formulation the textile swatches are dried on a washing line at ambient temperature.

Evaluation of micro creases

The creasing (surface smoothness) of the dried swatches is evaluated according to the AATCC-Standard method Nr. 124. Five persons evaluate the creases of the cotton swatches against the AATCC THREE DIMENSIONAL Smoothness Appearance Replicas. On the evaluation scale a number of 1 means very strong creasing, whereas 5 means almost no creasing.

Table 2 (Results of the evaluation of creases on cotton by AATCC method prior to ironing)

Sample of rinse conditioner formulation	AATCC evaluation
Reference	1.5
A	2.1
B	2.0
C	1.8

D	2.2
E	1.8
F	1.7
G	2.1
H	1.8
I	1.9
J	2.0
K	2.2
L	1.7
M	1.8
N	2.2
O	1.5
P	1.9
Q	2.0
R	1.8
S	2.4

These above results show a marked improvement in crease recovery for the textile fabric material treated with compositions of the present invention.

Example 3 (Reduction of micro creases on cotton (after ironing))

The textile swatches (cotton woven) from Example 2 are divided in 2 parts and one of it (with a size of 20 cm to 40cm) is slightly rewetted with 6.5 ml water (fine sprayed over the textile surface) and ironed without pressure for 60 seconds at 160°C.

The micro creases of the ironed swatches are evaluated according AATCC-Standard method Nr. 124 as described in Example 2.

Table 3 (Results of the evaluation of creases on cotton by AATCC method after ironing)

Sample of rinse conditioner formulation	AATCC evaluation
Reference	3.5
A	4.8
B	4.0

- 37 -

C	5.0
D	4.0
E	4.0
F	4.0
G	4.4
H	4.5
I	4.0
J	4.0
K	4.0
L	4.5
M	4.5
O	4.5
P	4.5
Q	4.5
R	4.0
S	4.0

These results show that microcreases can be removed significantly better by ironing when the textile fabric material is treated with compositions of the present invention.

Example 4 (Reduction of micro creases on cotton/polyester (prior to ironing))

The formulated rinse conditioners (see Table 1) are applied according to the following procedure:

Woven cotton/polyester swatches of size of 50 cm by 40 cm are washed and rinsed according to procedure described in Example 2.

Evaluation of micro creases

The creasing (surface smoothness) of the dried swatches is evaluated according to procedure described in Example 2.

Table 4 (Results of the evaluation of creases on cotton/polyester by AATCC method prior to ironing)

Sample of rinse conditioner formulation	AATCC evaluation
Reference	2.6
A	2.7
B	3.0
C	2.9
D	2.6
E	2.9
F	2.9
G	3.1
H	2.8
I	2.8
J	2.8
K	3.0
L	2.8
M	2.9
N	2.9
O	2.9
P	2.5
Q	2.5
R	3.2
S	2.8

The above results show a marked improvement in surface smoothness for the textile fabric material treated with compositions of the present invention.

Example 5 (Reduction of micro creases on cotton/polyester (after ironing))

The textile swatches (cotton/polyester woven) from Example 2 are divided in 2 parts and one of it (with a size of 20 cm to 40cm) is slightly rewetted with 6.5 ml water (fine sprayed over the textile surface) and ironed without pressure for 60 seconds at 160°C.

The micro creases of the ironed swatches are evaluated according AATCC-Standard method Nr. 124 as described in Example 2.

Table 5 (Results of the evaluation of creases on cotton/polyester by AATCC method after ironing)

Sample of rinse conditioner formulation	AATCC evaluation
Reference	4.0
A	4.5
B	4.5
C	4.3
D	4.5
E	4.5
F	4.5
G	5.0
H	4.5
I	5.0
J	4.5
K	4.5
L	4.5
M	5.0
N	5.0
O	4.5
P	4.5
Q	4.5
R	4.5
S	5.0

These results show that surface smoothness is significantly improved by ironing when the textile fabric material is treated with compositions of the present invention.

Example 6 (Reduction of wet soiling of cotton)Treatment of the textile material

Woven cotton swatches of size of 50 cm by 40 cm are washed together with ballast material (cotton and cotton/polyester) in a AEG Oeko Lavamat 73729 washing machine maintaining the washing temperature at 40°C. The total fabric load of 1 kg is washed for 15 minutes with 33 g of ECE Color Fastness Test Detergent 77 (Formulation January 1977, according to ISO 105-CO6). The rinse conditioner formulation as described in Table 1 is applied in the last rinse cycle at 20°C. After rinsing with the formulation the textile swatches are dried on a washing line at ambient temperature.

Soiling procedure

The treated swatches are cut to 5g pieces and then "soiled" for 20 minutes in a Linitest apparatus at 80°C with a solution of

0.1 g/l Carbon black (Corax N765)

0.3 g/l Nionionic Surfactant (Dobanol 91-10)

(Liquor ratio 50:1)

The soiled swatches are rinsed 30 seconds with tap water, spun and dried on a line at 60°C.

Washing out of soil

In a third step the soiled textile swatches are washed in a Linitest apparatus for 20 minutes with 3 g/l ECE Detergent at 80°C using a liquor ratio of 50:1. The washed swatches are rinsed for 30 seconds with tap water, spun and dried on a line at 60°C.

Evaluation of Wet Soiling

The lightness value Y measured with a Datacolor Spectrophotometer SF 500 is taken as a measure for the amount of soil deposited on the textile. Decreasing values of Y mean higher soil deposits on the textile.

The lightness value Y is measured after soiling of the swatches with carbon black and after washing out of the soil.

Table 6 (Results of lightness value measurements after soiling of the cotton swatches)
(Y value prior to soiling = 93.5)

Sample of rinse conditioner formulation	Y after soiling
Reference	63.2
A	81.2
B	70.6
C	79.0
D	72.1
E	70.9
F	77.3
G	70.5
H	71.4
J	82.3
K	71.3
L	77.6
P	78.9
Q	69.7
S	71.9

The results in Table 6 show an improved wet soil behaviour (less staining) of the textile fabric material treated with compositions of the present invention.

Table 7 (Results after wash out of soil)

Sample of rinse conditioner formulation	Y after wash out
Reference	64.4
A	81.3
B	70.3
C	80.0
D	73.6
E	73.0
F	77.6
G	72.2
H	74.0
J	82.6
K	72.4
L	78.3
P	79.4
Q	73.6
S	73.4

Results in Table 7 demonstrate that besides reduced wet soiling the treated textile release in a wash process the soil more readily compared to untreated materials.

Example 7 (Reduction of wet soiling of cotton/polyester)

In this example cotton/polyester 66/34 woven: 85 g/m², bleached, with resin finishing is treated, soiled and the soiled washed out according to the procedure described in Example 6. The evaluation of wet soiling is described in Example 6.

Table 8 (Results of lightness value measurements after soiling of the treated polyester/cotton swatches)

(Y value prior to soiling = 92.5)

Sample of rinse conditioner formulation	Y after soiling
Reference	53.0
A	64.9
B	76.7
C	71.7
D	68.5
E	68.4
F	76.6
G	65.4
H	75.6
J	64.1
K	77.3
L	76.3
N	63.6
P	69.2
Q	74.2
R	63.8
S	62.3

Results in Table 8 show that an improved wet soil behaviour (less staining) of polyester/cotton fabric material can be achieved when treated with compositions of the present invention.

In a washing process treated polyester/cotton fabric releases soil more readily than untreated fabric (Results in Table 9).

Table 9 (Results after wash out of soil)

Sample of rinse conditioner formulation	Y after wash out
Reference	58.5
A	68.2
B	77.8
C	73.8
D	72.3
E	71.2
F	78.2
G	66.7
H	77.6
J	67.8
K	79.8
L	77.2
N	67.0
P	73.0
Q	75.8
R	67.5
S	65.9

These results (Table 9) show an improvement in wet soil release for the textile fabric material treated with compositions of the present invention.

In all experiments textile materials treated as follows are used:

Cotton woven: 120 g/m², bleached, with resin finishing :

Cotton/Polyester 66/34 woven: 85 g/m², bleached.

Both textiles were finished with a resin according to Oekotex Standard 100:

30 g/l of modified dimethyloldihydroxyethylene urea (70% active material)

9 g/l Magnesiumchloride (with 6 H₂O)

- 45 -

padding with a pick-up of approximately 80%

Drying at about 110 - 120 °C in a oven followed by a 4 minute curing step at 145°C